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चर्मशोधन वनस्पति सामग्री — परीक्षण पद्धति

(पहला पुनरीक्षण)

Indian Standard

VEGETABLE TANNING MATERIALS — METHODS OF TEST

(First Revision)

ICS 59.140.10

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BUREAU OF INDIAN STANDARDS
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Leather, Tanning Materials and Allied Products Sectional Committee, CHD 17

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Leather, Tanning Materials and Allied Products Sectional Committee had been approved by the Chemical Division Council.

This standard was originally published in 1969. In this revision, a test method has been introduced to estimate pentachloro phenol (PCP) in vegetable tanning materials, keeping in view of the demand for eco-friendly inputs from the leather industry.

Vegetable tannins are polyphenols with molecular weight in the range of 500 to 3 000, present in vegetable tanning materials and in its extract. The materials are obtained in the form of wood, bark, fruit and leaves from various plants. The commonly available tanning materials in India are myrobalan nuts of the tree *Terminalia chebula* Retz., fam. Combretaceae; cutch extracts of the heartwood of the tree *KHAIR, Acacia catechu* (Linn.f.) Willd., fam. Leguminosae, A. *chundra* Willd., and *A. catechuoides* Benth; Wattle-bark of Acacia mearnsii De Willd. Syn. *A. mollissima* auct. non Willd., and *A decurrens* Willd; *Goran* bark of the tree *GORAN* [Ceriops decandra (Griff.) Ding Hou Syn. *Ceriops roxburghiana* Arn., fam. Rhizophoraceae], *AVARAM* bark of the shrub *Cassia auriculata* Linn, fam. Leguminosae; *BABUL* bark of the tree *BABUL* [Acacia nilotica (Linn.) Del. Syn. *A. arabica* Linn.] and *SONALI* bark of the tree *SONALI* (*Cassia fistula* Linn., fam. Leguminosae). The Committee while formulating these specifications thought it necessary to have a standard on the methods of test for determining the characteristics of various tanning materials.

The methods of test are based on the ‘Official methods of analysis’, published by the Society of Leather Traders’ Chemists, U. K. However, to suit Indian conditions, the temperature of cooling the infusion before testing has been changed from 18°C to 27 ± 2°C as this does not adversely affect the repeatability and reproducibility of the method.

The composition of the Committee responsible for the formulation of this standard is given in Annex A.

In reporting the result of a test or analysis made in accordance with this standard, If the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 ‘Rules for rounding off numberical values (*revised*)’.

Indian Standard

VEGETABLE TANNING MATERIALS — METHODS OF TEST

(First Revision)

1 SCOPE

1.1 This standard prescribes the methods of test for vegetable tanning materials of all kinds and their extracts.

1.2 This standard covers methods of test for the determination of moisture, total solid, total solubles, non-tannins, tannins, insoluble, *p*H colour, iron, copper and pentachloro phenol (PCP) content of tanning materials and their extracts.

2 REFERENCES

The standards listed below contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed below:

IS No.	*Title*
265 : 1993	Hydrochloric acid (*fourth revision*)
537 : 2007	Toluene, pure — Specification (*second revision*)
1070 : 1992	Reagent grade water (*third revision*)
1640 : 2007	Glossary of terms relating to hides, skins and leather (*first revision*)
2837 (Part 2) : 1977	Porcelain crucibles and basins: Part 2 Basins (*first revision*)
14575 : 1999	Determination of pentachlorophenol (PCP) in leather — Method of test

3 TERMINOLOGY

For the purpose of this standard, definitions given in IS 1640 and the following shall apply.

3.1 Total Solids — The amount of solid material contained in any tanning material. This is generally obtained by deducting the moisture content from the total mass of the material.

3.2 Total Solubles — The total amount of water soluble material present in any vegetable tanning material.

3.3 Insolubles — The amount of material present in any vegetable tanning material that are not soluble in water. In case of extract, this is obtained from the difference between the percentages of total solid and total solubles. But in other cases, in which moisture is determined directly, it is determined by the difference between 100 percent and the sum of the percentages of moisture and total solubles.

4 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and reagent grade water (*see* IS 1070) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the test results.

5 PREPARATION OF SAMPLE FOR TEST

5.1 Solid Crude Materials

Grind woods, barks, fruits, leaves or any other solid crude material in a suitable mill until it passes through a 1.40-mm IS sieve. Further separate the ground material by passing through a 600-micron IS sieve. Weigh the finer and coarser portions to determine the proportion of fine and coarse material in the ground sample and take aliquot portions of each grade for extraction.

NOTES

1 The apertures of BS sieve 12, ASTM sieve 14 (also known as 1.41 mm US standard sieve) and BS sieve 25, ASTM sieve 30 (also known as 595 microns US standard sieve) are within the limits laid down for 1.40-mm and 600-micron IS sieve respectively and may, therefore, be used in their place.

2 An 'Apex' Model 116A Cutter Mill, preferably fitted with a limit switch and brake, is most suitable for the purpose, but any mill producing a similar result may be used.

3 In the case of fibrous materials which after grinding leave a proportion of fibre incapable of passing through 1.40-mm IS sieve, the fibre portion shall be weighed separately, thus determining the proportion of three grades for analysis, namely, fibre, fine ground and coarse ground.

4 Fibrous materials may, where appropriate, be pounded in a mortar to break up the fibrous matter.

5 Since some materials lose moisture during grinding, it is advisable to determine moisture before and after grinding, and calculate the results on the ground sample back to the original moisture content.

5.2 Solid Extracts

Grind the solid extract in a porcelain mortar prior to weighing for analysis.

NOTE — In cases where the extract is of uneven moisture content and can be ground, the block should be broken up, a portion weighed in a flat-bottomed basin, allowed to dry in an oven at 70 ± 1°C for some hours and left exposed to the atmosphere overnight. After this partial drying, the extract is weighed and the loss of water calculated. The extract is next finely pulverized in a mortar and a weighed portion is dried in an oven at 98.5°C to 100°C to constant weight. This new loss of moisture is taken into consideration for calculating the percentage of water in the original sample.

5.3 Spray Dried Powder

Mix thoroughly and sample.

5.4 Liquid Extracts

Mix thoroughly so as to include any sediment which may be present in the liquid extract sample.

NOTE — In the case of viscous extracts, the sample should be heated to 45°C on a water bath, mixed, cooled to 27 ± 2°C and weighed at once, and the fact stated on the report.

6 PREPARATION OF INFUSION OF WATER SOLUBLE MATERIALS FROM SAMPLE OF GROUND WOODS, FRUITS, LEAVES AND OTHER TYPES OF TANNING MATERIALS

6.1 Quantity of Materials to Employ

Weigh out such a quantity of material as will give as nearly as possible 4.0 g/l of tannin matter absorbable by hide powder, and in any case not less than 3.75, nor more than 4.25 g.

NOTE — In the event of the results of an analysis showing a tanning strength outside these limits, the analysis shall be repeated employing the required amount of material.

6.2 Solid Crude Material

6.2.1 *Apparatus*

6.2.1.1 *Proctor extractor*, a beaker of tall shape and without spout is used; it shall be of 250 ml capacity for fresh materials and of 400 ml capacity for spent tans. The beaker is placed in a water bath. The bottom of the beaker is covered with a layer of clean sand 2 cm deep. For this purpose good quality sand is cleaned by washing thoroughly with hydrochloric acid, then with water and finally dried. A thistle funnel bent twice at right angles with the bell covered with a piece of well washed muslin is taken and the bell inserted in the layer of sand in the beaker. The stem is attached by a rubber connection bearing a screw clip, to a short glass tube which leads into the collecting flask. The bell of the thistle funnel shall be approximately 35 mm in diameter. The tanning material to be extracted is put into the beaker without disturbing the layer of sand and covered with distilled water. The further supply of water for the extraction comes from a conical flask of about 1 litre capacity which is also immersed in the water bath and which is itself supplied from a bottle standing at a convenient height above it. By regulation of the stopcock and the screw clip, the inflow and outflow can be so adjusted that the liquor in the beaker always stands at the same height, thus allowing the extraction to be carried out as required (*see* Fig. 1).

6.2.2 *Procedure*

Extract the ground sample (*see* **5.1**) in a Proctor extractor, using such a quantity as will give 2 litres of solution of the required analytical strength (*see* **6.1**). Soak the material in cold distilled water in the extractor overnight before commencing the extraction. Next morning draw off the infusion obtained, and continue the extraction at such uniform speed that 2 litres will be obtained in 4 h. After collecting the first 150 ml, raise the temperature to 50°C and collect a further 750 ml at this temperature. Then raise the temperature to boiling point as rapidly as possible, and extract the further quantity at this temperature to make to 2 litres.

NOTES

1 Woods shall be extracted so that the 2 litres will be secured by uniform extraction during 7 h instead of 4 h.

2 Materials yielding aqueous infusions that deposits ellagic or chebulinic acid shall not be soaked overnight in cold distilled water. The extraction of such materials shall be commenced at 50°C and after 1 litre has been extracted at this temperature, the extraction is completed as near the boiling point as possible.

6.2.3 *Cooling of the Infusion*

Immerse the flask in a large vessel (a sink is convenient) containing water at 27 ± 2°C and maintain the same temperature throughout the cooling process, agitating the flask from time to time. Continue the cooling until the temperature of the contents of the flask has reached 27 ± 2°C. Make up to the mark with water at the same temperature.

NOTES

1 It is essential that at no time during the cooling shall the temperature of the water in the cooling vessel fall below 27 ± 2°C in order to avoid local cooling at the walls of the flask.

2 Where it is difficult to maintain the temperature, the flasks, after cooling, may be placed in paper bags.

6.3 Solid Extract

Weigh out such a quantity of material as will give as nearly as possible 1 litre of solution of required analytical strength (*see* **6.1**). Wash into a beaker with boiling water, stir, and transfer the dissolved portion to a 1-litre flask. Repeat the process until all the extract is dissolved and the flask contains about 900 ml

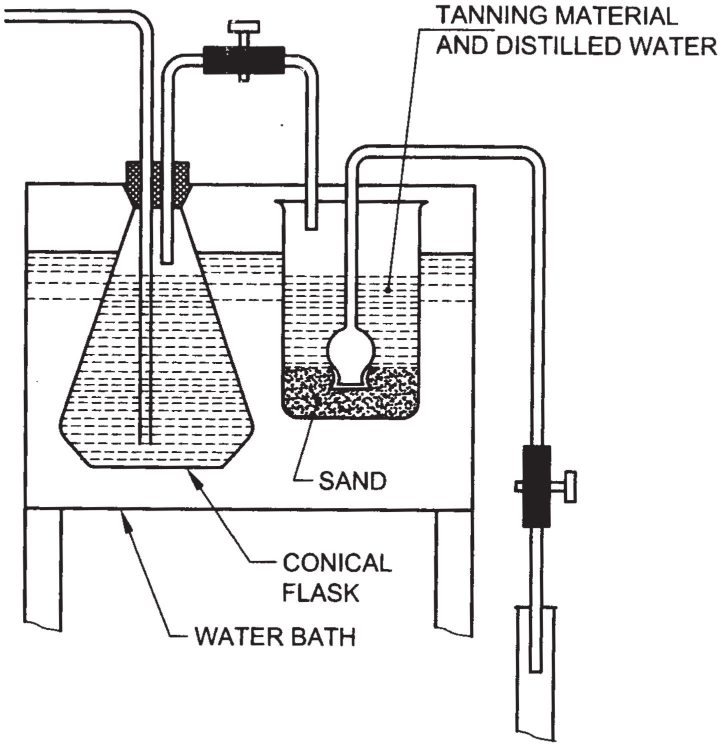

FIG. 1 PROCTOR EXTRACTOR

of solution. Cool and make up the volume as given in **6.2.3**.

NOTE — Extracts which are sensitive to boiling water should be dissolved at a lower temperature, and the temperature shall be stated on the report.

6.4 Liquid Extract

Weigh out such a quantity of material as will give as nearly as possible 1 litre of solution of required analytical strength (*see* **6.1**). Add 400 ml boiling water and transfer to a 1-litre graduated flask. Add further boiling water to bring the volume to about 900 ml. Cool and make up the volume as given in **6.2.3**.

7 DETERMINATION OF MOISTURE

7.1 Apparatus

7.1.1 *Vacuum or Air Oven,* with thermostatic control, capable of maintaining 100 ± 2°C.

7.1.2 *Wide-Mouth Weighing Bottle*

7.2 Procedure

Transfer about 2 to 5 g of the finely ground material (*see* **5**) into a tared wide-mouth weighing bottle and weigh accurately. Dry it at about 98.5 to 100°C in an oven for 3 to 4 h, cool in a desiccator for about 20 min and weigh again accurately. Repeat the process of, drying and weighing until two weighings at an interval of 1h do not differ by more than 2 mg.

NOTES

1 Since for any extract or material the sum of the moisture and the total solids is 100 percent, a determination of either is sufficient.

2 If the mass of the dried material is found to increase with longer drying, then the lowest mass found shall be taken.

7.3 Calculation

$$\text{Moisture, percent by mass} = \frac{(W_1 - W_2) \times 100}{W_1}$$

where

W_1 = mass of the material taken for the test, in g; and

W_2 = mass of the residue left after drying, in g.

8 DETERMINATION OF TOTAL SOLIDS

8.1 Apparatus

8.1.1 *Steam-Bath*

8.1.2 *Vacuum or Air Oven,* with thermostatic control, capable of maintaining 100 ± 2°C.

8.2 Procedure

Evaporate to dryness on a steam-bath 50 ml of the uniformly turbid tannin infusion prepared from a known amount of material according to method prescribed in **6.** Dry the residue at 98.5 to 100°C in an oven to constant mass. Consider the mass constant when two weighings at an interval of 1 h do not differ by more than 2 mg.

8.3 Calculation

$$\text{Total solid, percent by mass} = \frac{W_2}{W_1} \times 100$$

where

W_2 = mass of the material left after drying, in g; and

W_1 = mass of the material taken for test, in g.

9 DETERMINATION OF TOTAL SOLUBLES

9.1 Apparatus

9.1.1 *Berkefeld Candle*, 130 × 28 mm. New candles shall be cleaned by being left in concentrated hydrochloric acid for a month, then washed with water until the washings gives no acid reaction. Candles may also be washed by allowing hot water to pass through them. They are then immersed in hot dichromate-sulphuric acid mixture for 24 h, cleaned with water until there is no reaction for sulphate, and dried.

NOTE — Only those candles should be used whose porosity is in agreement with good tried candles.

9.1.2 *Filtration Apparatus,* as shown in Fig. 2 or Fig. 3.

9.1.3 *Porcelain Basin*, flat bottom, capacity 45 ml, conforming to IS 2837 (Part 2).

9.1.4 *Steam-Bath*

9.2 Procedure

Pour about 400 ml solution (as prepared in **6**) into a glass cylinder into which dips a clean and dry filter candle. Leave the candle immersed in tannin solution for about 10 min before starting the filtration. The necessary suction may be obtained by using a column of solution of 75 cm length (*see* Fig. 2) or by using a suction pump (*see* Fig. 3) keeping the vacuum fairly constant never exceeding 20 cm of mercury. Reject the first 250 ml of the filtrate and continue the filtration until it is clear to both transmitted and reflected light. Evaporate the next 50 ml of the filtrate in a porcelain dish to dryness and weigh. Repeat the process of drying and weighing until constant mass is obtained. If a clear filtrate cannot be obtained by the use of the candle, then mix 1 g of the kaolin/ 250 ml of the solution before filtration. Tanning solutions which are optically clear in the unfiltered state need not be filtered.

NOTE — In case Berkefeld candles are not available, the following procedure shall be adopted for filtration through fluted filter paper (Whatman No. 11/ No. 2 or Munketells No. 1 or equivalent is suitable). To 1 g kaolin in a beaker add sufficient solution to fill the filter paper, stir and pour on a filter paper. Return filtrate to paper when approximately 25 ml has been collected, repeating operation for 1 h, being careful to transfer all kaolin to the paper. At the end of one hour remove solution from filter paper, disturbing the kaolin as little as possible. Refill the paper with original solution and begin to collect the filtrate in a fresh beaker as soon as it comes optically clear. The paper shall be kept full and the funnels and collecting vessels shall also be covered at the time of filtration. Pipette 50 ml of the filtrate at 27± 2°C in a porcelain basin for evaporation, dry and weigh. Repeat the process of drying and weighing until constant mass is obtained.

9.3 Calculation

$$\text{Total solubles, percent by mass} = \frac{W_2}{W_1} \times \frac{V_1}{V_2} \times 100$$

where

W_2 = mass of the residue left after drying, in g;

V_1 = volume made up originally, in ml;

W_1 = mass of tanning material taken, in g; and

V_2 = volume of the test solution taken or pipetted out,in ml.

10 DETERMINATION OF NON-TANNINS

10.1 Principle

The non-tannins are determined from the matter left after the tannins are absorbed by hide powder.

10.2 Apparatus

10.2.1 *Glass Cylinder*, 500 ml capacity.

10.2.2 *Filtration Flask*, 500 ml capacity.

10.2.3 *Porcelain Basin,* between 7 to 8.5 cm in diameter, glazed inside conforming to IS 2837 (Part 2).

10.2.4 *Steam-Bath*

10.2.5 *Vacuum Oven,* with thermostatic control, capable of maintaining 100 ± 2°C.

10.3 Reagents

10.3.1 *Hydrochloric Acid* — Conforming to IS 265.

10.3.2 *Chrome Alum Solution* — Dissolve 30 g of chrome potassium sulphate, analytical grade, in distilled water at room temperature and make up the solution to 1 litre. Chrome alum solution more than 30 days old shall not be used.

10.3.3 *Kaolin* — Take such quality of kaolin that when 1 g of it is suspended in 100 ml of distilled water and well shaken, the *p*H of the suspension shall be between 4.0 and 6.0, that is, it shall neither give red colour with methyl orange nor deep purple colour with bromocresol purple. When 1 g of kaolin is shaken with 100 ml of 0.01 N acetic acid and the mixture filtered, the filtrate shall leave less than 1 mg of residue after evaporation and drying.

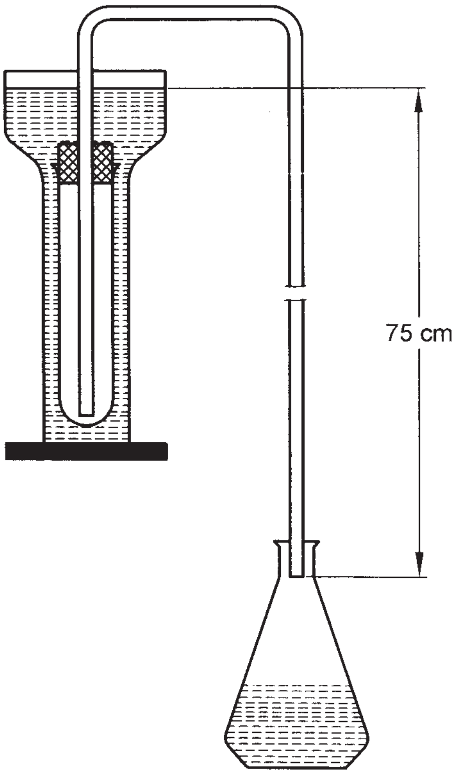

FIG. 2 FILTRATION APPARATUS

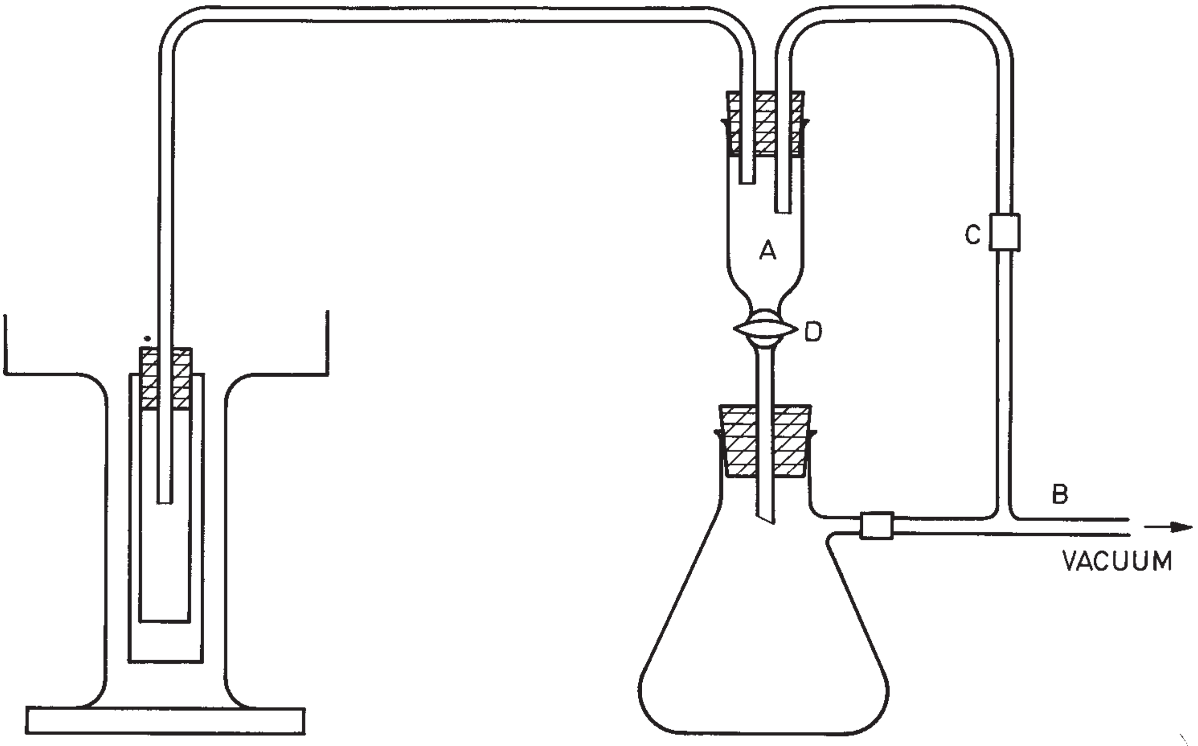

FIG. 3 APPARATUS FOR FILTRATION UNDER REDUCED PRESSURE

10.3.4 *Chromed Hide Powder* — For each analysis digest a multiple of that quantity of hide powder containing 6.25 g of dry matter, with ten times its mass of distilled water for 1 h. Add to this 1 ml of chrome alum solution for each gram of air-dry hide powder taken, stir frequently for several hours and then allow to stand overnight. Next morning transfer the chromed powder to a clean linen, drain and squeeze. Place the cloth containing the powder in a suitable vessel (an enamel bucket is suitable for large quantities), open out the cloth bag and pour on to the powder a quantity of water equa1 to 15 times the mass of the air-dry hide powder taken. Mix the powder and water thoroughly and digest for 15 min, after which lift out the cloth and powder and immediately drain and squeeze the powder so that it contains approximately 75 percent moisture. Digest the powder three times more in the same way with distilled water. Thoroughly break up the cake of chromed powder, mix until uniformly free from lumps and weigh the whole.

10.3.5 *Linen* — Pieces of linen cloth shall be used for washing the chromed hide powder and for the preliminary filtration of detannized solutions. The linen shall be freed from weighting matter by boiling in several changes of distilled water.

10.3.6 *Hide Powder* — It shall comply with the following requirements:

a) The ash content of the powder shall be not more than 0-3 percent, by mass.
b) When 7 g of the air-dry powder is allowed to stand in contact with 100 ml of 0.1 N potassium chloride solution, previously adjusted to *p*H 5.5 with 0.01 N acetic acid, for 24 h then the liquor obtained by centrifuging or by filtering through a paper previously washed with 0.1 N potassium chloride solution, shall have a *p*H ranging between 5.0 and 5.5.

10.3.7 *Gelatin-Salt Reagent* — Dissolve 1 g of photographic grade gelatin and 10 g of pure sodium chloride in 100 ml of distilled water at a temperature not higher than 60°C and adjust the *p*H to 4.7 approximately by adding acid or alkali, that is, the solution shall give red colour with methyl red and yellow with methyl orange. To preserve this solution, add 2 ml toluene (conforming to IS 537) or preferably, a few crystals of *p*-dichlorobenzene and keep in a cool place. Freshly prepared solution is preferable.

10.4 Procedure

Weigh accurately a quantity of wet hide powder (say X g) containing about 6.25 g dry powder (*see* **10.3.4**) and add it immediately to 100 ml of the unfiltered tannin infusion (prepared as in **6**) plus $(26.25 - X)$ ml of distilled water, already present in a wide mouth bottle of 150 to 300 ml capacity. Stopper the wide mouth bottle tightly with a rubber bung, and shake it vigorously first by hand for 15 s and then transfer to a mechanical rotary shaker and shake for exactly 10 min at 50 to 65 rev/min. Pour powder and solution on a clean, dry linen filter cloth supported by a funnel, drain and squeeze by hand. Add to the filtrate 1 g of kaolin and pour into a single 15 cm pleated filter paper (Whatman No. 11/No. 2 or Munketelle's No. 1F or equivalent is suitable) returning the filtrate repeatedly until it is clear. Keep the funnel and collecting vesel covered during filtration. Test the filtrate with gelatin-salt reagent and if 10 ml gives any turbidity with 1 or 2 drops of the reagent, mention this in the report of the analysis. Evaporate in a tared porcelain dish 50 ml of the filtrate and dry the residue in a vacuum oven at 98.5° to l00°C, cool and weigh until constant mass is obtained.

NOTE — To correct for the 20 ml of water of dilution introduced by the wet hide powder into the 100 ml of tannin solution, the residue weight is multiplied by 1.2. This corrected weight is the residue from 60 ml of original analytical solution.

10.5 Calculation

$$\text{Non-tannins, percent by mass} = \frac{W_2}{W_1} \times \frac{V_1}{V_2} \times 100$$

Where

W_2 = mass of the residue left after drying, in g;
V_1 = volume made up originally, in ml;
W_1 = mass of the material taken, in g; and
V_2 = volume of the test solution taken, in ml.

11 DETERMINATION OF TANNINS

11.1 Principle

The tannins are determined by finding the difference between the percentages of total solubles and the non-tannins.

11.2 Procedure

11.2.1 Determine the total solubles of the tannin material according to the method given in **9**.

11.2.2 Determine the non-tannins present in the tanning material according to the method given in **10**.

11.3 Calculation

Calculate the tannin as follows:

$$\text{Tannins, percent by mass} = X - Y$$

where

X = total solubles, percent by mass (*see* **11.2.1**); and
Y = non-tannins, percent by mass (*see* **11.2.2**).

NOTES

1 All analysis should be done in duplicate. The absolute error in tannin content in duplicate analysis should not be more than 2 percent.

2 Where analysis are carried out by different chemists on the same sample, the result should not differ by more than 3 percent of the total tannin content.

12 DETERMINATION OF INSOLUBLES

12.1 Principle

In the case of extracts, insolubles are determined from the difference between the percentages of total solids and total solubles. But in other cases, where moisture is determined directly, it is determined by the difference between 100 percent and the sum of the percentages of moisture and total solubles.

12.2 Procedure

12.2.1 Determine the total solid (in case of extracts only) according to the method prescribed in **8.**

12.2.2 Determine the total solubles according to the method prescribed in **9.**

12.2.3 Determine moisture (in case of non-extracts) in accordance with the method prescribed in **7.**

12.3 Calculation

Calculate the insolubles as follows.

12.3.1 *In Case of Extracts*

Insolubles, percent by mass = $X - Y$

where

X = total solids, percent by mass (*see* **12.2.1**); and

Y = total solubles, percent by mass (*see* **12.2.2**).

12.3.2 *For Materials Other Than Extracts*

Insolubles, percent by mass = $100 - (X + Y)$

where

X = moisture content (*see* **12.2.3**); and

Y = total solubles, percent by mass (*see* **12.2.2**).

13 DETERMINATION OF *p*H

13.1 Apparatus

13.1.1 p*H Meter*, with glass electrode.

13.2 Procedure

Determine the *p*H of the solution prepared as in **6,** adjusting the relative density to 105 g/ml at 27°C with cold water, by using a suitable *p*H meter.

14 DETERMINATION OF COLOUR

14.1 Apparatus

14.1.1 *Lovibond Tintometer,* with artificial light source (A 60-W Osram Pearl lamp, or its equipment is the suitable artificial light source and such lamp should be replaced after 200 hours' use).

14.2 Procedure

Fill the solution prepared in **6** immediately after filtration in a 1-cm cell and determine the colour value. Calculate the result on 0.5 percent tannin content basis and express in units of red and yellow.

15 DETERMINATION OF IRON

15.1 Reagents

15.1.1 *Fuming Nitric Acid*

15.1.2 *Bromine Water* — Saturated.

15.1.3 *Concentrated Hydrochloric Acid* — Conforming to IS 265.

15.1.4 *Ammonium Hydroxide* — 1:1 (*v/v*).

15.1.5 *Dilute Hydrochloric Acid* — 1:1 (*v/v*).

15.1.6 *Dilute Sulphuric Acid* — I : 20 *(v/v*).

15.1.7 *Potassium Permanganate Solution* — Approximately 0.1 N.

15.1.8 *Potassium Thiocyanate Solution* — Dissolve 10 g of potassium thiocyanate (or ammonium thiocyanate) in 100 ml of water.

15.1.9 *Stock Iron Solution* — Dissolve exactIy 0.702 g of pure ferrous ammonium sulphate [$FeSO_4 (NH_4)_2 SO_4 . 6H_2O$] in 50 ml of water and 20 ml of dilute sulphuric acid (1:4) and add 0.1 N potassium permanganate solution drop by drop until a faint pink colour is obtained. Adjust the volume to one litre with water. The strength of this solution is ten times that of the standard solution. The keeping quality of this solution is better at this strength. Store this solution and use it when required.

15.1.10 *Standard Iron Solution* — Prepare a fresh solution by diluting 10 ml of the stock solution (*see* **15.1.9**) to 100 ml with water. Each millilitre of this solution contains 0.000 01 g of iron (as Fe).

15.2 Procedure

15.2.1 Weigh accurately a quantity of material which is equivalent to 5 g on moisture free basis, to the nearest 0.1 mg in a glazed flat bottomed porcelain basin. Heat it first over a low flame until the residue is completely charred and ceases to give out smoke. Then place the basin in an electric muffle furnace (approximately 600°C) to make it free of carbon.

15.2.2 In case all the carbon is not removed, take out the basin from the furnace and allow it to cool. Add a little hot water and break up the carbonaceous matter with a glass rod. Extract it with water by warming it over a water bath for a few minutes. Decant the supernatant liquid, filter it through an ashless filter paper and collect the filtrate. Repeat the process of extraction and filtration thrice. Combine the filtrates. Transfer the residue on the filter paper and wash it several times with hot water and add the washings to the filtrate. Place the filter paper and the contents in the original basin, dry, ignite and ash as before, until all the carbon is burnt off. Cool the basin again and transfer the combined filtrates and washings quantitatively to it. Evaporate the extract to dryness on a water bath. Replace the basin in an electric muffle furnace (approximately 600°C).

15.2.3 Cool the basin and moisten the carbon-free ash with hot water. To this add five drops each of fuming nitric acid and bromine water. Heat slowly until the fumes cease to come out. Evaporate to dryness. Moisten the residue with water and digest it with 5 ml of concentrated hydrochloric acid over a water bath for a few minutes, dilute with water and filter into a 250-ml beaker. Adjust the volume to approximately 75 ml (by boiling, if necessary) and make it faintly alkaline with ammonium hydroxide. Boil off the excess ammonia gently and allow the precipitated iron and aluminium hydroxides to coagulate.

15.2.4 Allow the solution to stand for few minutes until the precipitate settles. Decant the supernatant liquid quickly through an asbestos Gooch filter previously washed with hot dilute hydrochloric acid, water, ammonium hydroxide and water successively. Wash the precipitate three times with hot water made faintly alkaline with five drops of ammonium hydroxide per litre. Transfer the precipitate quantitatively to the filter and wash it four times with hot faintly ammonical water. Keep the combined filtrate and washings for the determination of copper (*see* **16**).

15.2.5 Dissolve the precipitate on the filter paper by treating it with two lots of 20 ml dilute sulphuric acid and collect the solution. Wash the filter paper four times with dilute sulphuric acid and combine the washings with the solution. Add a few drops of potassium permanganate solution until the solution is slightly pink and transfer it to a 250-ml volumetric flask. Adjust the volume finally to the mark with dilute sulphuric acid.

15.2.6 Transfer 10 ml of dilute sulphuric acid and 5 ml of potassium thiocyanate solution to each of the two identical 50-ml Nessler cylinders. To one of the Nessler cylinders add 10 ml of the made up solution (*see* **15.2.5**) and make up the volume to 50 ml mark. From a burette, run in standard iron solution to the second Nessler cylinders until the tints of the two cylinders are identical when viewed vertically through the depths of the cylinders, the cylinders being held over a white surface.

15.3 Calculation

Calculate the iron content of the sample from the known volume of the standard iron solution required for matching.

16 DETERMINATION OF COPPER

16.1 Apparatus

16.1.1 *Nessler Cylinder* -— 50 ml capacity.

16.2 Reagents

16.2.1 *Tetra-Sodium Pyrophosphate Solution* — 4 percent.

16.2.2 *Gum Arabic* — 1 percent solution.

16.2.3 *Sodium Diethyldithiocarbamate Solution* — Dissolve 1.0 g of sodium diethyldithiocarbamate in 500 ml of copper-free water. Filter and keep in an amber bottle and protect from light.

16.2.4 *Stock Copper Solution* -— Dissolve exactly 3.928 3 g of copper sulphate crystals ($CuSO_4.5H_2O$) in water and make up the volume to one litre.

16.2.5 *Standard Copper Solution* — Prepare a fresh solution by diluting 10 ml of the stock solution (*see* **16.2.4**) to one litre with water and adjust the *p*H value between 5.5 and 6.0. Each millilitre of this solution contains 0.000 01 g of copper (as Cu).

16.3 Procedure

16.3.1 Heat the combined filtrate and washings preserved in **15.2.4** until the free ammonia has been driven off, cool and make up to 100 ml. Measure an aliquot, usually 10 ml, into a 50-ml Nessler cylinder, together with 10 ml of tetrasodium pyrophosphate and 5 ml of gum arabic solution, mix carefully, add 5 ml of sodium diethyldithiocarbamate and make up to volume with distilled water.

16.3.2 To another Nessler cylinder add 10 ml of tetrasodium pyrophosphate, 5 ml of gum arabic solution, 5 ml of sodium diethyldithiocarbamate and water almost up to volume. From a burette, run in standard copper solution until the yellow colour in the first cylinder is matched, when viewed vertically through the depth of solutions, the cylinders being held vertically over a white surface.

NOTES

1 The volume of standard copper solution required should not exceed 6 ml.

2 With extracts containing relatively large amounts of copper, it is advantageous to make up the solution after separation of the iron to a greater volume than 100 ml. This obviates the necessity of measuring very small aliquots.

3 Tetrasodium pyrophosphate solution is used to sequester manganese, calcium and magnesium salts which may give rise to opalescence.

4 Separation of the coloured copper complex is prevented by means of the gum arabic solution.

16.4 Calculation

Calculate the copper content of the sample from the known volume of the standard copper solution required for matching.

17 DETERMINATION OF PENTACHLORO PHENOL (PCP)

17.1 Vegetable Tanning Materials

The determination of PCP is to be carried out in the same manner in IS 14575. Five grams of the vegetable tanning material is to be taken in the place of leather sample. However a *p*H check to ascertain the nature of the sample is to be done before the extraction. In case the sample is alkaline in nature, then after weighing and transferring the sample into extraction thimble, wet the same thoroughly with 10-15 percent *W/V* aqueous sulphuric acid (a simple wetting is sufficient. Care is to be taken to avoid adding excess of aqueous acid which would generate a pool of aqueous layer leading the poor extraction). Add about 5-7 g of anhydrous sodium sulphate along with the sample. Then follow the procedure as given in Method A of IS 14575.

17.2 Vegetable Tanning Extracts

17.2.1 Transfer about 5 g of the vegetable tanning extract into a separating funnel of about 250 ml capacity along with 100 ml water. Add 15-25 ml of sulphuric acid 10 percent (*W/V)* aqueous solution and ensure that the *p*H of the contents is acidic. Then carry out liquid – liquid extraction using hexane, analytical grade, as the solvent. At first extract with about 25 ml hexane by shaking the contents thoroughly for about 15 min. Then set it aside for clear separation of the layers (*see* Note below). Transfer the aqueous layer into another separating funnel and carryout three more extractions of the aqueous layer with hexane of 25 ml each time in the same manner explained earlier.

NOTE — In case of any emulsion being noticed, break the same with the addition of 20-30 ml saturated sodium chloride aqueous solution or follow the steps explained in **4.3.4.2** of IS 14575.

17.2.2 Combine the hexane extracts and dry it by passing through a bed of anhydrous sodium sulphate of about 10 g. The dried hexane layer is evaporated to near dryness by a rotary evapourator.

17.2.3 The residue is taken for acetylation. Then follow the procedure as detailed in Method A of IS 14575.

18 REPORTING THE RESULTS

18.1 The results shall be reported to one place of decimals only.

18.2 All analysis shall be made strictly in accordance with the methods prescribed and the report shall state,

a) that the analysis has been made according to this standard;
b) the batch number of hide powder;
c) the method of extraction employed;
d) whether the tannin infusion has been filtered or not;
e) whether kaolin has been used for the filtration of the tanning infusion;
f) whether moisture has been determined by direct drying; and
g) if water at a temperature lower than boiling point is used for the preparation of the analytical solution (*see* **6.3**), the temperature shall be stated.

ANNEX A

(*Foreword*)

COMMITTEE COMPOSITION

Leather, Tanning Materials and Allied Products Sectional Committee, CHD 17

Organization	*Representative(s)*
Central Leather Research Institute, Chennai	Director (***Chairman***)
A V Thomas Leather & Allied Products Pvt Ltd, Chennai	Shri Habib Hussain Shir K. Manivannan (*Alternate*)
All India Skins & Hide Tanners and Merchants Assciation, Chennai	Shri Mohan M. Sreenivas Shri S. Mohammed Hassan (*Alternate*)
BASF India Ltd, Mumbai	Shri P. R. Chaudhari Dr S. A. Nadgouda (*Alternate*)
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Central Footwear Training Institute, Agra	Shri S. N. Ganguly Shri S. Chakraborty (*Alternate*)
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Consumer Federation of India, New Delhi	Representative
Council for Leather Exports, Chennai	Shri M. M. Hashim Dr Zackria Sait (*Alternate*)
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International Institute of Saddlery Technology and Export Management, Kanpur	Representative
Italab Private Ltd, Chennai	Shri M. A. Etiappan Shri J. J. Patel (*Alternate*)
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Organization	*Representative(s)*
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Scientist 'F' and Head (CHD), BIS

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